

- I. Instrument Name: Aspex Scanning Electron Microscope/Energy Dispersive X-ray Analysis System
- II. Suggested Uses: High Resolution and magnification imaging with enhanced depth of field for trace evidence. This system provides non-destructive elemental analysis of gunshot residue particles, paint, metal, powders, and other trace particulate material.
- III. Operating Procedures
- A. Start-up and Analysis
1. Turn instrument on
 - a. Double click on **Perception icon** to bring up the SEM screen on left monitor.
 - b. Click on **Power button** (Turns bright green when Powder is on).
 - c. Saturate the beam and align Filament at 20kv Accelerating Voltage (Use 10KV for first time if using a new filament)
 - i. Adjust the **Filament Drive** up to around 68% or until an image appears on **Right** monitor.
 - ii. Turn filament drive up slowly until the image is at maximum brightness.
 - iii. Select Source Imaging Mode from the drop down menu on the Right monitor to view the emission image.
 - iv. Adjust Filament Drive until the emission image is slightly under saturation.
 - v. Select Line Scan option from the drop down menu on the Right monitor.
 - vi. Adjust the X and Y axis control on the LEFT monitor to adjust the line scan on the Right monitor until the line scan is at maximum height.
 - vii. Close the line screen mode on the Right monitor as well as the x and y gun alignment controls on Left monitor.
 - d. Adjust the accelerating voltage to the desired KV setting for the particular analysis. Gunshot Residue will require 20KV accelerating voltage.
 - e. Choose variable Pressure Option. High vacuum mode is used for all occasions except items that may charge. Variable pressure is used for the non-coated charging samples. Default variable pressure setting is 0.1 Torr.
 2. Run Sample
 - a. Load the sample(s) into the specimen holder and identify the location of each sample on the appropriate stage position drawings.
 - b. Vent the specimen chamber of the microscope (if not already vented) and load new samples into microscope.

- i. Turn Beam Filament **OFF** before venting.
- ii. Move black handle or lever on the stage/chamber from the downward position towards the UPWARD position. This will begin the process of venting the chamber.
- iii. The handle will automatically move to the full UP position once the chamber is at atmospheric pressure.
- iv. Once the chamber has dropped down, PULL the stage chamber out from the microscope column.
- v. Loosen the thumb screw on the right side of the stage mount.
- vi. Remove the holder from the stage mount.
- vii. If using the same stage mount, remove the already analyzed samples, place them into appropriate vials, and load samples as described in 2a section of this document.
- viii. Insert the newly loaded specimen holder back into the stage mount and tighten the thumb screw on the mount.
- ix. Lift stage/chamber assembly and gently slide it back into the open cavity of the microscope.
- x. Push the black handle down to begin pumping out the chamber (placing microscope under vacuum).
- xi. When system is at Vacuum Ready, the Power button on upper left corner of Left monitor will be a dull green.
- f. Saturate the beam at the desired KV setting (20KV for most elemental applications and all Gunshot Residue Analysis)
- g. For non- Gunshot Residue Analysis applications, follow this protocol:
 - i. Confirm the following instrumental parameters to be suitable or change for particular applications: magnification range, spot size, backscattered or secondary electron detector (For our work, backscattered is usually sufficient and preferred), High vacuum or variable pressure mode, and scan speeds.
 - ii Center particles of interest in the middle of the yellow box (The zoomed area will be visible on the right side of right monitor.)
 - ii Focus the particles and click on the particle to initiate collecting an X-ray spectra.
 - iii Edit the X-ray setup(including the selection of elements of interest, display parameters, and labeling of peaks).
 - iv Visually inspect and confirm the entire spectra to confirm presence of elements and correct labeling of peaks.
 - v. Save the spectra, image, or both.
 - vi. Generate the analysis report using one of the already

formatted templates or individually formatted templates generated by each operator.

- e. Gunshot Residue Analysis
 - i. Uses a specific program already loaded into the software in the Aspex.
 - ii. Identify the sample name and information for each adhesive lift in the sample holder.
 - iii. Recheck the three stage positions for each adhesive lift and store these points along with the correct focus or Working distance for each lift.
 - iv. Adjust X-ray dead ime on Copper standard to 20-30%. Adjust brightness and contrast on Copper standard using the Threshold icon to a lower setting of 16 and upper setting of 185.
 - v. Choose the location for storing the data from this analysis.
 - vi. Check the preferences for the analysis and choose the appropriate one----Shutting the beam off after analysis and returning to High vacuum after analysis is completed (if Variable pressure mode is used) are two of the options most often selected.
 - vii. Press GO to start the analysis.
 - viii. Review the results after the automated analysis has finished
 - (a) Report icon shows list of particles, Relocate icon automatically returns stage to the field where the particle is located.
 - (b) Center particle, focus it, adjust brightness/contrast, perform elemental analysis on particle, confirm elements present and proper labels, and press SAVE icon on GSR section of the left screen to save new image and new x-ray spectra.
 - (c) Review all particles of interest.
 - (d) Press Report on the GSR section of the left monitor to generate the particle report for all of the confirmed particles.

B. Shut down procedures

- 1. Ensure that the X-ray system is not actively collecting X-ray spectra.
- 2. Turn filament drive to 0%.
- 3. Press Power button again to switch power to OFF. Bright green light will become dull green..
- 4. Power off the two monitors.
- 5. If shutting system down for longer periods.
 - a. Follow steps 1-4 listed above.. Then close out Perception.
 - b. Then Click on Start icon in lower left corner of the desk top.

- c. Then select Shut down to shut the computer off.
 - C. Calibration
 - 1. Calibration of the X-ray system will be performed a minimum of once per month. However, calibration is not limited to once per month and can be performed as needed.
 - 2. Calibration procedures can be found in the Trace Evidence Section Calibrations Manual.
- IV. Safety Concerns
 - A. Exposure to x-ray radiation
 - 1. EDS x-rays are considered to be “soft” and of relatively little health risks.
 - 2. Monitor badges are located near the system to monitor the radiation from the EDS.
 - B. Exposure to Liquid Nitrogen
 - 1. Liquid nitrogen is used to cool the detector of the EDS.
 - 2. Prolonged exposure to Liquid Nitrogen can be harmful to the skin and the eyes.
 - 3. It is recommended to use Safety insulated gloves and eye protection when filling the dewar of the EDS system manually.
 - C. High voltage exposure
 - 1. The SEM/EDS system operates on a 110 Volt power outlet. However, high voltage is generated within the system and applied to the filament assembly in order to produce the electron beam.
 - 2. Therefore, an electrical shock hazard can exist during certain maintenance procedures. Use caution when dealing with high voltage supplies.

Note: Aspex SEM/EDS manuals, logs, and reference materials are located in the SEM suite near the user station. Manuals and reference material are also located on the computer (See Desktop page).